PATENT COOPERATION TI ATY

To:

| From the | INTERNATIONAL | BUREAU |
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PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

Commissioner

US Department of Commerce United States Patent and Trademark Office, PCT 2011 South Clark Place Room

CP2/5C24 Arlington, VA 22202

ETATS-UNIS D'AMERIQUE in its capacity as elected Office

Date of mailing (day/month/year) 30 January 2001 (30.01.01)

International application No.

PCT/FI00/00542

International filing date (day/month/year)

15 June 2000 (15.06.00)

Applicant's or agent's file reference

VAI 81 PCT

Priority date (day/month/year) 17 June 1999 (17.06.99)

Applicant

STORMBOM, Lars et al

| 1. | The designated Office is hereby notified of its election made: |
|----|---|
| | X in the demand filed with the International Preliminary Examining Authority on: |
| | 17 November 2000 (17.11.00) |
| | in a notice effecting later election filed with the International Bureau on: |
| | |
| 2. | The election X was |
| | was not |
| | made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b). |
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The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland

Authorized officer

R. E. Stoffel

Telephone No.: (41-22) 338.83.38

Facsimile No.: (41-22) 740.14.35



PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

| Applicant's or agent's file reference VAI 81 PCT | FOR FURTHER ACTION | 1 | eation of Transmittal of International v Examination Report (Form PCT/IPEA/416) |
|---|--|---------------------------|---|
| International application No. | International filing date (day | month year) | Priority date (day/month/year) |
| PCT/FI00/00542 | 15.06.2000 | | 17.06.1999 |
| International Patent Classification (IPC) o | r national classification and IP | C ₇ | |
| G 01 N 27/02, G 01 N | 27/22, G 01 N 3 | 3/28 | |
| · | | | · |
| Applicant | | • | |
| VAISALA OYJ ET AL | | | |
| VAIDABA CTO ET AE | | | |
| This international preliminary exa Authority and is transmitted to the | e applicant according to Article | 2 36. | |
| 2. This REPORT consists of a total of | of 4 sheets, inc | uding this cover | · sncet. |
| been amended and are the b | | ts containing rec | ion, claims and/or drawings which have tilications made before this Authority the PCT). |
| These annexes consist of a total o | f 3 sheets. | | |
| 3. This report contains indications re | lating to the following items: | | |
| I Basis of the report | | | |
| II Priority | | | |
| III Non-establishment of | opinion with regard to novelt | v, inventive step | and industrial applicability |
| IV Lack of unity of inver | ntion | | |
| | mder Article 35(2) with regard tions supporting such statemen | | entive step or industrial applicability; |
| VI Certain documents cit | | | |
| VII Certain defects in the | international application | | |
| VIII Certain observations | on the international application | ı | |
| نسا | | | |
| | | | |
| Date of submission of the demand | Date | of completion | of this report |
| 17.11.2000 | 07 | .09.2001 | |
| Name and mailing address of the IPEA/SE | C Aut | norized officer | |
| Patent- och registreringsverket Box 5055 | Tele:: 17078 | | |
| s-102 42 sтоскноы; Facsimile No. 08-667_72_88 | FATOREGES JO | nas Ande phone No. 08- | rsson/LR |

Form PCT/IPEA/409 (cover sheet) (January 1998)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

| International application No. |
|-------------------------------|
| PCT/FI00/00542 |

| J. B | asis of the report | |
|-------------------|---|---|
| 1. Wi | th regard to the elements of the international application:* | |
| Г | the international application as originally filed | |
| $\bar{\boxtimes}$ | the description: | • |
| L | pages 1-10 | , as originally filed |
| | | , filed with the demand |
| | pages | , filed with the letter of |
| \geq | the claims: | |
| | pages | , as originally filed |
| | pages | , as amended (together with any statement) under article 19 |
| | pages | , filed with the demand |
| abla | 7 | , filed with the letter of 24.08.2001 |
| | the drawings: | as originally filed |
| | pages | , as originally filed |
| | pages | , filed with the letter of, filed with the demand |
| | the sequence listing part of the description: | · |
| <u> </u> | pages | , as originally filed |
| | pages | , filed with the demand |
| | pages | , filed with the letter of, filed with the demand |
| the | th regard to the language, all the elements marked above wer international application was filed, unless otherwise indicates se elements were available or furnished to this Authority in t | |
| | the language of a translation furnished for the purposes of | international search (under Rule 23.1(b)). |
| | the language of publication of the international application | n (under Rule 48.3(b)). |
| | the language of the translation furnished for the purposes or 55.3). | of international preliminary examination (under Rules 55.2 and/ |
| | h regard to any nucleotide and/or amino acid sequence dis- liminary examination was carried out on the basis of the sequ | |
| | contained in the international application in written form. | |
| | filed together with the international application in comput | er readable form. |
| | furnished subsequently to this Authority in written form. | |
| | furnished subsequently to this Authority in computer read | able form. |
| | The statement that the subsequently furnished written sequenterinational application as filed has been furnished. The statement that the information recorded in computer representation been furnished. | nence listing does not go beyond the disclosure in the readable form is identical to the written sequence listing has |
| 4. | The amendments have resulted in the cancellation of: | |
| | the description, pages | |
| | the claims, Nos. | |
| | the drawings, sheet/fig | |
| 5. | | Iments had not been made, since they have been considered to go nental Box (Rule 70.2 (c)).** |
| in t | | Office in response to an invitation under Article 14 are referred to |
| | v replacement sheet containing such amendments must be ref | ferred to under item I and annexed to this report. |

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/FI00/00542

| V. | Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; |
|----|--|
| | citations and explanations supporting such statement |

| 1. | Statement | | | |
|----|-------------------------------|------------------|------|--------|
| | Novelty (N) | Claims Claims | 1-14 | YES NO |
| | Inventive step (IS) | Claims Claims | 1-14 | YES NO |
| | Industrial applicability (IA) | Claims Claims | 1-14 | YES NO |

2. Citations and explanations (Rule 70.7)

The invention relates to a method and an apparatus measurement of water content of a liquid by simultaneously performing two different electrical measurements. Properties measured using both a relative-value liquid are measurement and an absolute-value measurement. The apparatus of the invention comprises one sensor sensitive to changes in dielectric coefficient and another sensitive to sensor relative water content.

The invention is intended to solve the problem concerning determination of water content in oil, which usually require a zeroing step that is temperature dependent.

The method according to amended claims 1-8 further comprises repetition of the measurement at two different succession at a constant water temperatures in a rapid content. The apparatus according to amended claims 9-14 also that the sensor means adapted for measuring the relative water content contains an auxiliary medium capable of absorbing water contained in the liquid under measurement. These features are not revealed by the prior art documents cited in the International Search Report and there is no hint in these documents leading a person skilled in the art to the solution according to present invention.

Consequently, the invention according to amended claims 1-14 is novel, industrially applicable, and is considered to involve an inventive step.

International application No.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

PCT/FI00/00542

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

The independent claim 1 is not considered to fulfil the requirement on clarity and conciseness. Expressions like "a property of the liquid is measured electrically for one parameter" and "another electrical method" are too broad and do not define how the invention is actually performed. Further, the expression "measurement method resulting in the measurement of the liquid for its dielectric coefficient and relative water content" of claim 1 is directed to a result to be obtained without clearly defining how this is actually done.

Claim 1 is further unclear since it presents the wish that "said measurement is repeated at two different temperatures in a so rapid succession that the water content of the liquid may be assumed to stay at least substantially constant". This expression is vague and does not give a clear definition on how rapid the measurement should be repeated.

Consequently, the subject matter of claim 1 should be clarified in order to define all the features needed to obtain the result in view.

ne Swedish Patent Office
Uninternational Application

What is claimed is:

1. Method for measurement of water content of a liquid, in which method a property of the liquid is measured electrically for one parameter,

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- the properties of the liquid are measured at least substantially simultaneously also by another electrical method, whereby the properties of the liquid are measured using both a relative-value measurement method and an absolute-value measurement method resulting in the measurement of the liquid for its dielectric coefficient and relative water content

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characterized in that

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 said measurement is repeated at two different temperatures in a so rapid succession that the water content of the liquid may be assumed to stay at least substantially constant.

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2. Method according to claim 1, c h a r a c t e r i z e d in that the temperature dependence of the dielectric coefficient of an entirely dry liquid is determined by measuring essentially simultaneously the ε_r and temperature of the liquid at at least two temperatures.

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- 3. Method according to claim 1 or 2, c h a r a c t e r i z e d in that said relative water content measurement is carried out using a capacitive sensor.
- 4. Method according to any one of foregoing claims, characterized in that changes in the results of water content measurement due to aging of the liquid are compensated for by virtue of using only the most recent data of the measurement history for the compensation for changes in the response of the measurement system.

- 5. Method according to any one of foregoing claims, characterized in that the aging of said liquid, advantageously oil, is indicated on the basis of changes in the value of ε_0 .
- 6. Method according to any one of foregoing claims, characterized in that, in the measurement of relative water content, an auxiliary medium is used for absorbing thereto the water contained in the liquid under measurement.

Method according to claim 5, characterized in that said auxiliary medium is a thin-film polymer layer.

8. Method according to claim 5 or 6, c h a r a c t e r i z e d in that the water content of said auxiliary medium is determined by way of measuring its dielectric coefficient.

Apparatus for measurement of the water content of a liquid, said apparatus comprising one electrical sensor means (5, 6 or 1, 6) for measuring the water content of a liquid, which apparatus includes

- a second electrical sensor means (1, 6 or 5, 6) for measuring the water content of a liquid, said second sensor means (1, 6 or 5, 6) measuring a different parameter than that measured by said first electrical sensor means (5, 6 or 1, 6), said sensor means being such that one of them measures the properties of the liquid by a relative-value measurement method and the other by an absolute-value measurement method, whereby one sensor means (5, 6) is sensitive to changes in the dielectric coefficient and the other sensor means is sensitive to the relative water content,

characterized in that

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the sensor means adapted for measuring the relative water content-contain an auxiliary medium capable of absorbing water contained in the liquid under measurement.

10. Apparatus according to claim 9, c h a r a c t e r i z e d in that said sensor means (5, 6) sensitive to changes in dielectric coefficient is formed by two interdigitated finger electrodes (5, 6).

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11. Apparatus according to claim 9 or 10, c h a r a c t e r i z e d in that one electrode (6) of the sensor pair adapted to perform the measurement of dielectric coefficient also forms a part of the measurement electrode pair (1, 6) adapted to perform the measurement of the relative water content.

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- 2. Apparatus according to claim 9, c h a r a c t e r i z e d in that the sensor means sensitive to changes in the dielectric coefficient is formed by a coaxial structure, wherein one electrode is formed by a center pin and the jacket has a net-like structure and is permeable to water.
- 13. Apparatus according to claim 12, characterized in that said auxiliary medium is a thin-film polymer layer.

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14. Apparatus according to claim 12 or 13 characterized in that the apparatus contains means adapted to measure the dielectric coefficient of said auxiliary medium whereupon the relative water content of said auxiliary medium can be determined.

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International application No. PCT/FI 00/00542

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: G01N 27/02, G01N 27/22, G01N 33/28
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE, DK, FI, NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

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| UU | OC, | 77 | PΙ |

| L1 0000 | 1111 4 | |
|-----------|--|-----------------------|
| C. DOCU | MENTS CONSIDERED TO BE RELEVANT | |
| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
| х | WO 9923469 A1 (MULTI-FLUID INC.), 14 May 1999 (14.05.99), page 2, line 15 - line 17; page 3, line 24 - line 30; page 13, line 1 - line 21, page 16, line 14 - line 18 | 1-2,4-5,9-11 |
| A | | 3, 6-8,12-15 |
| | ~- | |
| A | US 5331287 A (FREDERID G. YAMAGISHI ET AL), 19 July 1994 (19.07.94) | 6-8,10,13-15 |
| | | |
| A | US 4129501 A (EDWARD M. HAYNES), 12 December 1978 (12.12.78) | 6-8,13-15 |
| | | |
| | · | |
| X Furth | er documents are listed in the continuation of Box C. X See patent family annex | ι |

| <u></u> , | | | |
|--|--|--|--|
| "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand | | | |
| the principle or theory underlying the invention | | | |
| "X" document of particular relevance: the claimed invention cannot be | | | |
| considered novel or cannot be considered to involve an inventive step when the document is taken alone | | | |
| "Y" document of particular relevance: the claimed invention cannot be | | | |
| considered to involve an inventive step when the document is combined with one or more other such documents, such combination | | | |
| being obvious to a person skilled in the art | | | |
| "&" document member of the same patent family | | | |
| Date of mailing of the international search report | | | |
| 26 October 2000 (26.10.00) | | | |
| | | | |
| Authorized officer | | | |
| | | | |
| Jonas Andersson/GH | | | |
| Telephone No. + 46 8 782 25 00 | | | |
| : | | | |

International application No.
PCT/FI 00/00542

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Information on patent family members

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International application No.
PCT/FI 00/00542

| Patent do cited in sear | | | Publication date | | tent family member(s) | Publication date |
|----------------------------|---------|----|------------------|----------------------|---|--|
| WO : | 9923469 | A1 | 14/05/99 | AU | 1121799 A | 24/05/99 |
| US ! | 5331287 | A | 19/07/94 | DE EP JP JP | 69326726 D,T 0584557 A,B 2610775 B 6160319 A | 29/06/00 02/03/94 14/05/97 07/06/94 |
| US | 4129501 | A | 12/12/78 | NONE | | |
| US | 5644239 | A | 01/07/97 | WO | 9528645 A | 26/10/95 |
| EP (| 0141636 | A1 | 15/05/85 | GB GB ZA | 2149117 A 8329483 D 8408489 A | 05/06/85 00/00/00 26/03/86 |
| WO ! | 9846984 | A2 | 22/10/98 | EP | 0975955 A | 02/02/00 |
| DE 1 | 9647201 | C1 | 12/02/98 | NONE | | |

RECORD COPY

PCT REQUEST

Original (for SUBMISSION) - printed on 15.06.2000 08:47:36 AM

VAI 81 PCT

| 0 | For receiving Offic use only | |
|------------|---|---|
| 0-1 | International Application No. | PCT/FI 0 0 / 0 0 5 4 2 |
| 0-2 | International Filing Date | 1 5 JUN 2000 (1 5 -86- 2000) |
| 0-3 | Name of receiving Office and "PCT International Application" | The Finnish Patent Office PCT International Application |
| 0-4 | Form - PCT/RO/101 PCT Request | T |
| 0-4-1 | Prepared using | PCT-EASY Version 2.90 (updated 10.05.2000) |
| 0-5 | Petition | (aparon notice) |
| 0-5 | The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty | |
| 0-6 | Receiving Office (specified by the | National Board of Patents and |
| | applicant) | Registration (Finland) (RO/FI) |
| 0-7 | Applicant's or agent's file reference | VAI 81 PCT |
| ī | Title of invention | METHOD AND APPARATUS FOR MEASURING WATER |
| | | CONTENT |
| II II-1 | Applicant This person is: | applicant only |
| II-2 | Applicant for | all designated States except US |
| 11-4 | Name | VAISALA OYJ |
| II-5 | Address: | P.O. Box 26 |
| | | FIN-00421 Helsinki |
| | | Finland |
| II-6 | State of nationality | FI |
| 11-7 | State of residence | FI |
| 11-8 | Telephone No. | +358-9-894 91 |
| 11-9 | Facsimile No. | +358-9-894 9227 |
| 111-1 | Applicant and/or inventor | |
| 111-1-1 | This person is: | applicant and inventor |
| III-1-2 | Applicant for | US only |
| III-1-4 | Name (LAST, First) | STORMBOM, Lars |
| III-1-5 | Address: | Raappavuorenreuna 2 A 2 |
| | | FIN-01620 Vantaa |
| | | Finland |
| III-1-6 | State of nationality | FI |
| 111-1-7 | State of residence | FI |

VAI 81 PCT

Original (for SUBMISSION) - printed on 15.06.2000 08:47:36 AM

| III-2 | Applicant and/or inventor | |
|------------------|--|--|
| III-2-1 | This person is: | applicant and inventor |
| 111-2-2 | Applicant for | US only |
| 111-2-4 | Name (LAST, First) | LYYRA, Matti |
| 111-2-5 | Address: | Aamutie 3 B 2 |
| | | FIN-01670 Vantaa |
| | | Finland |
| 111-2-6 | State of nationality | FI |
| 111-2-7 | State of residence | FI |
| 111-3 | Applicant and/or inventor | |
| III-3-1 | This person is: | applicant and inventor |
| III-3 - 2 | Applicant for | US only |
| 111-3-4 | Name (LAST, First) | LEPPÄNEN, Jukka |
| 111-3-5 | Address: | Kasavuorentie 1 as. 1 |
| | | FIN-02700 Kauniainen |
| | | Finland |
| 111-3-6 | State of nationality | FI |
| 111-3-7 | State of residence | FI |
| IV-1 | Agent or common representative; or | |
| | address for correspondence The person identified below is | agent |
| | hereby/has been appointed to act on | agec |
| | behalf of the applicant(s) before the competent International Authorities as: | |
| IV-1-1 | Name | SEPPO LAINE OY |
| IV-1-2 | Address: | Itämerenkatu 3 B |
| | | FIN-00180 Helsinki |
| | | Finland |
| IV-1-3 | Telephone No. | +358-9-68 59 560 |
| IV-1-4 | Facsimile No. | +358-9-68 595 610 |
| IV-1-5 | e-mail | seppo.laine@selpat.fi |
| V | Designation of States | |
| V-1 | Regional Patent (other kinds of protection or treatment, if | EP: AT BE CH&LI CY DE DK ES FI FR GB GR |
| | any, are specified between parentheses | IE IT LU MC NL PT SE and any other State |
| | after the designation(s) concerned) | which is a Contracting State of the |
| | | European Patent Convention and of the |
| | | PCT |
| V-2 | National Patent | JP US |
| | (other kinds of protection or treatment, if any, are specified between parentheses | |
| | after the designation(s) concerned) | |

Original (for SUBMISSION) - printed on 15.06.2000 08:47:36 AM

| V-5 | Precautionary D signation Stat ment | | |
|---|---|---|---|
| | In addition to the designations made | | |
| | under items V-1, V-2 and V-3, the | | |
| | applicant also makes under Rule 4.9(b) | | |
| | all designations which would be | | |
| | permitted under the PCT except any | | |
| | designation(s) of the State(s) indicated | | |
| | under item V-6 below. The applicant | | |
| | declares that those additional | | |
| | designations are subject to confirmation | | |
| | and that any designation which is not | | |
| | confirmed before the expiration of 15 | | |
| | months from the priority date is to be | | |
| | regarded as withdrawn by the applicant at the expiration of that time limit. | | |
| V-6 | Exclusion(s) from precautionary | NONT | |
| | designations | NONE | |
| VI-1 | Priority claim of earlier national application | | |
| VI-1-1 | Filing date | 17 June 1999 (17.06. | 1999) |
| VI-1-2 | Number | 991391 | |
| VI-1-3 | Country | FI | |
| VI-2 | Priority document request | | |
| | The receiving Office is requested to | VI-1 | |
| | prepare and transmit to the International | | |
| | Bureau a certified copy of the earlier | | |
| | application(s) identified above as | | |
| | item(s): International Searching Authority | Swedish Patent Offic | (TCA/CE) |
| | | | |
| VII-1 | | Swedish Patent Offic | e (ISA/SE) |
| VII-1 | Chosen Check list | number of sheets | electronic file(s) attach d |
| | Chosen | | |
| VIII | Chosen Check list | number of sheets | electronic file(s) attach d |
| VIII VIII-1 | Chosen Check list Request | number of sheets | electronic file(s) attach d |
| VIII VIII-1 VIII-2 | Chosen Check list Request Description | number of sheets 4 | electronic file(s) attach d - |
| VIII VIII-1 VIII-2 VIII-3 | Chosen Check list Request Description Claims | number of sheets 4 8 3 | electronic file(s) attach d |
| VIII VIII-1 VIII-2 VIII-3 VIII-4 | Chosen Check list Request Description Claims Abstract | number of sheets 4 8 3 1 1 1 | electronic file(s) attach d vai81pct.txt |
| VIII VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 | Chosen Check list Request Description Claims Abstract Drawings | number of sheets 4 8 3 1 | electronic file(s) attach d vai81pct.txt |
| VIII VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 | Chosen Check list Request Description Claims Abstract Drawings TOTAL | number of sheets 4 8 3 1 1 1 | electronic file(s) attach d vai81pct.txt - |
| VIII VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 VIII-7 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items | number of sheets 4 8 3 1 1 17 paper document(s) attached | electronic file(s) attach d vai81pct.txt - electronic file(s) attached |
| VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 VIII-7 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items Fee calculation sheet | number of sheets 4 8 3 1 1 17 paper document(s) attached | electronic file(s) attach d vai81pct.txt - electronic file(s) attached - |
| VIII VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 VIII-7 VIII-8 VIII-16 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items Fee calculation sheet PCT-EASY diskette | number of sheets 4 8 3 1 1 17 paper document(s) attached Copy of official | electronic file(s) attach d vai81pct.txt - electronic file(s) attached - diskette |
| VIII VIII-1 VIII-2 VIII-3 VIII-5 VIII-7 VIII-8 VIII-16 VIII-17 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items Fee calculation sheet PCT-EASY diskette Other (specified): | number of sheets 4 8 3 1 1 17 paper document(s) attached - Copy of official action | electronic file(s) attach d vai81pct.txt - electronic file(s) attached - diskette |
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| VIII VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 VIII-7 VIII-8 VIII-16 VIII-17 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items Fee calculation sheet PCT-EASY diskette Other (specified): Figure of the drawings which should accompany the abstract Language of filing of the international application | number of sheets 4 8 3 1 1 17 paper document(s) attached Copy of official action 1 | electronic file(s) attach d vai81pct.txt - electronic file(s) attached - diskette |
| VIII-1 VIII-2 VIII-3 VIII-5 VIII-7 VIII-16 VIII-17 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items Fee calculation sheet PCT-EASY diskette Other (specified): Figure of the drawings which should accompany the abstract Language of filing of the international | number of sheets 4 8 3 1 1 17 paper document(s) attached Copy of official action 1 | electronic file(s) attach d vai81pct.txt - electronic file(s) attached - diskette |
| VIII VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 VIII-7 VIII-8 VIII-16 VIII-17 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items Fee calculation sheet PCT-EASY diskette Other (specified): Figure of the drawings which should accompany the abstract Language of filing of the international application | number of sheets 4 8 3 1 1 17 paper document(s) attached Copy of official action 1 | electronic file(s) attach d vai81pct.txt - electronic file(s) attached - diskette |
| VIII VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 VIII-7 VIII-8 VIII-16 VIII-17 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items Fee calculation sheet PCT-EASY diskette Other (specified): Figure of the drawings which should accompany the abstract Language of filing of the international application | number of sheets 4 8 3 1 1 17 paper document(s) attached Copy of official action 1 | electronic file(s) attach d vai81pct.txt - electronic file(s) attached - diskette |
| VIII VIII-1 VIII-2 VIII-3 VIII-4 VIII-5 VIII-7 VIII-8 VIII-16 VIII-17 VIII-18 VIII-19 | Chosen Check list Request Description Claims Abstract Drawings TOTAL Accompanying items Fee calculation sheet PCT-EASY diskette Other (specified): Figure of the drawings which should accompany the abstract Language of filing of the international application Signature of applicant or agent | number of sheets 4 8 3 1 1 17 paper document(s) attached | electronic file(s) attach d vai81pct.txt - electronic file(s) attached - diskette |

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| 10-1 | Date factual rec ipt of th | 1 5 JUN 2000 | (15-06-2000) |
|------|-------------------------------------|---------------|----------------|
| | purp rted internati nal applicati n | 1 2 3014 2000 | |

(D 7. 07. DN)

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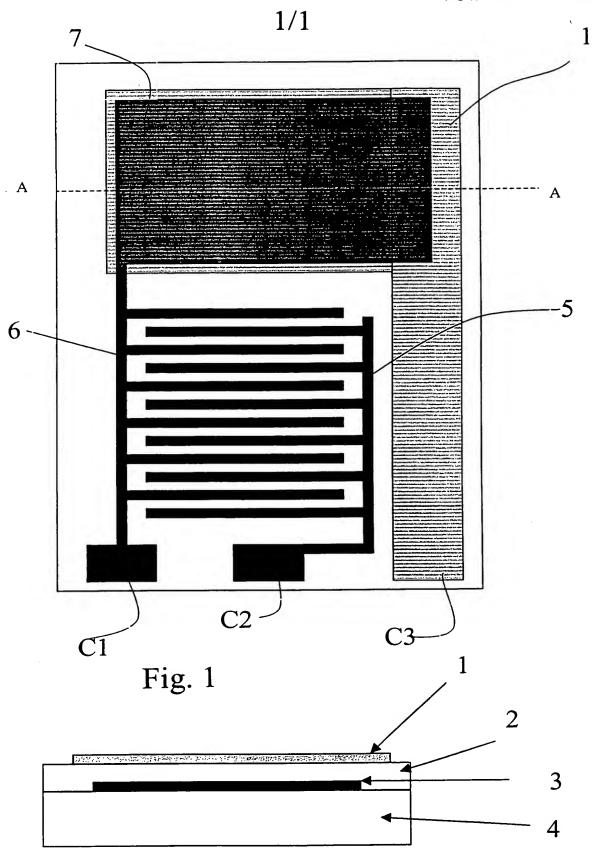


Fig. 2

Menetelmä ja laitteisto nesteen vesipitoisuuden mittausta varten

Keksinnön kohteena on patenttivaatimuksen 1 johdannon mukainen menetelmä nesteen vesipitoisuuden mittausta varten.

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Keksinnön kohteena on myös laitteisto nesteen vesipitoisuuden mittausta varten.

US-patentissa 5331287 kuvataan sensori, jossa interdigitoidut elektrodit (sormielektrodit) on päällystetty johdepolymeerillä. Öljyssä oleva vesi hydraa polymeerin ja muuttaa sen johtavuutta. Havaitsee myös mahdolliset hapot protonoinnin kautta.

US-patentissa 5642098 kuvataan rengasoskillaattorikytkentä, jossa useamman, samaa parametria mittaavan mittapään avulla mitataan öljyn sähköisiä ominaisuuksia.

US-patentissa 5644239 mitataan nesteen (öljyn) sähkönjohtavuus kahdessa korotetussa lämpötilassa. Lisäksi mitataan mahdollisesti optisesti öljyn "opacity" (sameus). Näiden parametrien avulla lasketaan öljyn "laatuluku".

US-patentissa 5656767 kuvataan sensorijärjestelmä, jossa mitataan öljyn sähköisen suureen (esim. kapasitanssin) muutosta ajan funktiona. Referenssisuureena voidaan käyttää puhdasta (kuiva) samaa öljyä. Monia variaatiota samasta teemasta jossa esim. lämmitetään näytettä yms.

Tunnetussa tekniikassa on useita puutteellisuuksia. Yleisiä koko alueen 0-100 % absoluuttitilavuusvesipitoisuuden mittausmenetelmiä ovat dielektrisyysvakion mittaus ja IR-absorption mittaus. Yhteistä molemmille menetelmille on se, että niissä tarvitaan mittarin nollaus, ts. täytyy säätää mittaus näyttämään 0-pitoisuutta kun anturissa on täysin kuivaa (vedetöntä) nestettä. Tämä voidaan hoitaa joko kertaluontoisena säätönä tai pitämällä sensorissa täysin kuivaa vertailuöljyä.

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Lisävaikeutena on että tämä 0-säätö tyypillisesti on lämpötilasta riippuva.

0-arvo voi myös muuttua muun kun veden vaikutuksesta nesteen vanhetessa.

Absoluuttimenetelmät ovat vahvoilla kun vesipitoisuudet ovat isoja (%-luokassa).

Pienillä pitoisuuksilla ongelmana on yleensä mittausherkkyys ja offset-epämääräisyydet (nollaus-virhe).

Suhteellisella (aw-) menetelmällä saadaan tieto vesipitoisuudesta verrattuna saturaatiotilaan. Muunnos absoluuttiseen volyymiprosenttiin on kuitenkin tuntematon ellei k.o. nesteen muunnoskerrointa ole määritetty. Aw-menetelmä on toimiva matalissa vesipitoisuuksissa (ei saturoitunut tila, ei emulsiotila), joissa saavutetaan riittävä mittausherkkyys. Myöskään nollausvirhettä ei ole.

Tämän keksinnön tarkoituksena on poistaa edellä kuvatun tekniikan puutteellisuudet ja aikaansaada aivan uudentyyppinen menetelmä ja laitteisto nesteen vesipitoisuuden mittausta varten.

Keksintö perustuu siihen, että mitataan öljyn/nesteen vesipitoisuutta samanaikaisesti kahdella eri menetelmällä, jolloin mittausmenetelminä käytetään absoluuttimenetelmää ja suhteellista mittausmenetelmää.

Täsmällisemmin sanottuna keksinnön mukaiselle menetelmälle on tunnusomaista se, mikä on esitetty patenttivaatimuksen 1 tunnusmerkkiosassa.

Keksinnön mukaiselle laitteistolle on puolestaan tunnusomasta se, mikä on esitetty patenttivaatimuksen 9 tunnusmerkkiosassa.

Keksinnön avulla saavutetaan huomattavia etuja.

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Yhdistämällä absoluuttimenetelmään suhteellinen menetelmä (aw-mittaus) voidaan poistaa absoluuttimenetelmän nollauksen tarve.

Käyttämällä nopeasti toistettuja mittauksia eri lämpötiloissa, voidaan myös lämpötilan muutoksista aiheutuvat virheet eliminoida.

Keksintöä ryhdytään seuraavassa lähemmin tarkastelemaan oheisten kuvioiden mukaisten suoritusesimerkkien avulla.

5 Kuvio 1 esittää yläkuvantona yhtä keksinnön mukaista elektrodirakennetta.

Kuvio 2 esittää leikkausta A-A kuvion 1 mukaisesta anturirakenteesta.

Kuvion 1 ja 2 mukaisesti keksinnön mukainen ratkaisu on toteutettavissa rakenteella, jossa substraatin 4 pinnalle on muodostettu kolme elektrodia. Itse substraatin 4 pinnalla on alaelektrodipari 3, joka on muodostettu elektrodeista 5 ja 6. Tässä tapauksessa elektrodit ovat ns. sormielektrodit eli interdigitoidut elektrodit, jossa mahdollisimman suuri elektrodien välinen pinta on muodostettu elektrodirakenteella, joka vastaa toistensa lomissa olevia sormia. Elektrodien 5 ja 6 toisiaan lähinnä olevien reunojen pituus vastaa tasokondensaattorirakenteen elektrodipinnan pinta-alaa. Elektrodien 5 ja 6 leveys samoin kuin niiden välinen matka (gap) on tyypillisesti 5-500 mikrometriä. Sormirakenteen lisäksi elektrodissa 6 on tasomainen alue 7. Elektrodit 5 ja 6 käsittävät myös kontaktialueet C1 ja C2 elektrodien kytkemiseksi edelleen mittalaitteisiin. Alaelektrodiparin 3 päälle on muodostettu polymeerikalvo 2, jonka paksuus on tyypillisesti 0,5-5 mikrometriä. Polymeerikalvo 2 voi ulottua myös sormielektrodirakenteen 5 ja 6 päälle, jolloin se toimii passivointina ja vähentää nesteen johtavien partikkelien vaikutusta mittaukseen. Polymeerikalvon 2 päällä on vettä läpäisevä yläelektrodi 1, jonka kontaktipisteenä toimii kontakti C3. Yläelektrodi 1 on sijoitettu elektrodin 6 suorakaidemaisen yhtenäisen alueen 7 päälle tasokondensaattorirakenteen muodostamiseksi.

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Kuvioiden 1 ja 2 rakennetta käytetään seuraavasti. Nesteen dielektrisyysvakio mitataan käyttäen elektrodien 5 ja 6 kontakteja C1 ja C2. Suhteellinen mittaus puolestaan toteutetaan elektrodien 6 ja 1 välillä, siis kontakteista C1 ja C3.

Neste, jonka vesipitoisuutta mitataan on edullisesti öljyä, mutta myös muiden nesteiden kuten hydrauliikkanesteen, bensiinin tai jäähdytysnesteen vesipitoisuutta voidaan mitata keksinnön mukaisesti.

Keksinnön mukaisesti vedenaktiviteettimittaus voidaan toteuttaa seuraavasti.

Vedenaktiviteettimittaus on vain yksi esimerkki keksinnön mukaisesta suhteellisen pitoisuuden mittauksesta.

Anturi voi olla esim. kuvioiden 1 ja 2 mukaisesti kahden elektrodin välillä oleva polymeerikalvo 2, joka absorboi vettä ympäristön vesiaktiviteetin funktiona. Tällaisia antureita ovat esim. suhteellisen kosteuden anturit. Tyypillistä tällaiselle mittausmenetelmälle on että mittauksen tuloksena saadaan veden aktiviteetti ts:

$$aw=F(ppm/ppm_s(T))$$
, missä (1)

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ppm=veden tilavuusosuus liuoksessa*106

ppm,=veden tilavuusosuus kylläisessä liuoksessa *106

15 Funktio F voidaan esim. olettaa lineaariseksi jolloin:

$$aw=ppm/ppm_s(T)$$
 (2)

aw vaihtelee välillä 0 (täysin kuiva neste) ... 1 (kylläinen vesiliuos).

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Pelkällä vedenaktiviteettimittauksella saadaan tieto siitä kuinka lähellä ollaan emulsio- tai vapaan veden tilaa. Kun emulsio- tai vapaan veden tila on saavutettu, vedenaktiviteetti on kuitenkin hyvin lähellä arvoa l jolloin nesteen tilasta ei saada informaatiota. Mittausperiaatteella saadaan kuitenkin pitoisuuksissa aw < 0,9 hyvin herkkä öljy/nestetyypistä riippumaton tieto esim. sen voiteluominaisuuksista.

Huonelämpötilassa ppm_s voi vaihdella välillä 20 ppm lisäaineistamattomalle perusöljylle aina yli 10000 ppm:n asti runsaasti lisäaineistetulle voiteluöljylle.

Dielektrisyysvakiomittausta kuvataan seuraavassa tarkemmin. Dielektrisyysvakiomittaus on vain yksi esimerkki keksinnön piiriin kuuluvista absoluuttipitoisuusmittauksista.

Nesteen dielektrisyysvakiomittauksessa voi anturi olla keksinnön mukaisesti interdigitoitu elektrodi (sormielektrodi) kuvioiden 1 ja 2 mukaisesti tai koaksiaalinen elektrodipari, joka on kosketuksissa mitattavan nesteen kanssa. Anturista saatava signaali on riippuvainen sekä mitattavan nesteen dielektrisyysvakiosta että sen vesipitoisuudesta:

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$$\varepsilon_r = \varepsilon_0 + F(ppm)$$
, missä (3)

 ϵ_0 =Täysin kuivan nesteen dielektrisyysvakio

F(ppm)=veden määrästä riippuva funktio. Voidaan rajoitetulla pitoisuusalueella olettaa lineaariseksi eli:

$$\varepsilon_r = \varepsilon_0 + a * ppm, missä$$
 (4)

15 a = nestetyypistä riippumaton vakio

Dielektrisyysmittauksen hyvä puoli on se, että se kattaa koko mahdollisen mittausalueen 0-100 tilavuus % vettä. Haittapuolena mittausmenetelmässä on se, että herkkyys kuivassa päässä (joka yleensä on kiinnostavin) on pieni ja että ϵ_0 on tunnettava. Tyypillisesti mittaus kalibroidaan käyttämällä kuivattua öljyä.

Jos molempia mittausmenetelmiä käytetään samanaikaisesti voidaan toimia useammalla tavalla nesteen kalibroimiseksi:

25 Mikäli nesteen ppm_s(T) tunnetaan, toimitaan seuraavasti:

Mitataan samanaikaisesti aw ja ε_r . Jos aw on alle 1 voidaan olettaa yhtälöiden 2 ja 4 pätevän.

Yhdistämällä yhtälöt 2 ja 4 saadaan ratkaistua ε₀ ja ppm. ppm_s(T) voidaan myös arvioida, jos nestetyyppi on tunnettu, jolloin saadaan jonkun verran epätarkempi tulos.

Mikäli nesteen ppm_s(T) on tuntematon, toimitaan seuraavasti:

Mitataan vasteet kahdella eri (tuntemattomalla) vesipitoisuudella. Jos molemmissa tapauksissa aw on alle 1 saadaan yhtälöryhmä, jossa on neljä yhtälöä ja neljä tuntematonta eli ratkaisu on yksikäsitteinen. Jos merkitään mittaustuloksia mittauskerroilla 1 ja 2 vastaavilla alaindekseillä saadaan:

$$\varepsilon_0 = (aw_2 * \varepsilon_{r1} - aw_1 * \varepsilon_{r2})/(aw_2 - aw_1)$$
 (5)

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Tämä toimenpide voi tapahtua myös automaattisesti mittalaitteen toimiessa jos mitattavan nesteen kosteuspitoisuus vaihtelee.

Jos dataa kerätään enemmän kuin kaksi mittausparia, voidaan tuntemattomat termit sovittaa dataan esim. pienimmän neliösumman menetelmää käyttäen.

Jälkimmäisen menetelmän vahvuutena on, että lisäksi että mahdolliset muutokset ϵ_0 :ssa tai ppm $_s(T)$:ssa lämpötilamuutosten tai nesteen vanhenemisen tai likaantumisen vuoksi voidaan kompensoida. Öljyn ikääntymistä voidaan indikoida ϵ_0 :n muutoksesta.

- Jos öljynäytteen lämpötilaa muutetaan tai näytevirtauksen lämpötilaa muutetaan niin nopeasti, että voidaan olettaa vesipitoisuuden pysyvän likimain vakiona voidaan määrittää täysin kuivan nesteen dielektrisyysvakion lämpötilariippuvuus mittaamalla lähes samanaikaisesti ε_r ja nesteen lämpötila ainakin kahdessa lämpötilassa.
- 25 Esimerkiksi voimme olettaa ε_0 olevan lineaarinen lämpötilan funktio:

$$\varepsilon_r = b0 + b1 * T + a * ppm$$
 (6)

tällöin voimme muodostaa

$$\varepsilon_{r}(T2) - \varepsilon_{r}(T2) = b1*(T2-T1)$$
 (7)

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josta on b1-kerroin ratkaistavissa. Tässäkin tapauksessa voi käyttää useampaa lämpötilapistettä ja pienimmän neliösumman menetelmää. Tällä tavalla saadaan jatkuvasti luotettava arvio ϵ_0 -parametrille lämpötilan muuttuessa.

5 Samanaikaisesti voidaan määrittää myös ppm_s(T):n lämpötilariippuvuus. Yleisesti voidaan olettaa rajatulla lämpötila-alueella:

$$ppm_s(T) = c0 * e^{(C1 * T)}$$
 (8)

10 Jossa

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c0= ppm_s:n arvo kun T=0°C c1=lämpötilariippuvuuskerroin

Tällöin voidaan mittaamalla aw ainakin kahdessa lämpötilassa ratkaista c1 oletuksella ettei nesteen vesipitoisuus muutu:

$$c1=LN(aw1/aw2)/(T2-T1)$$
 (9)

20 Edullisesti nämä kaksi määritystä voidaan suorittaa samanaikaisesti.

Keksinnön yhden edullisen suoritusmuodon mukaisesti suoritetaan nollaus automaattisesti aina kun aw-mittaus antaa riittävän pienen arvon. Mitä pienempi aw-arvo on sitä tarkempi nollaus saadaan. Epävarmuus menetelmässä liittyy ppm_s(T):n arvoon joka on nestetyypistä riippuva ja "arvattava".

Keksinnön toisen edullisen sovellusmuodon mukaisesti on käytettävissä kaksi näytettä nesteestä/öljystä eri vesipitoisuuksilla. Joko näytetään laitteelle tietoisesti kaksi eri vesipitoisuudella olevaa näytettä tai kerätään prosessista vähitellen dataa ja käytetään prosessissa tapahtuvaa luonnollista vesipitoisuuden vaihtelua. Saamme arvon myös ppm_s(T):lle jolloin voimme laskea keksinnön mukaisesti aw-arvosta myös ppm vesipitoisuuden, joka ei perustu "arvaukseen".

Jos kerätään dataa jatkuvasti prosessista ja samalla "unohdetaan" dataa vanhemmasta päästä, voidaan kompensoida myös nesteen/öljyn vanhenemisesta johtuvia muutoksia sekä ppm_s .:ssä että ϵ_0 :ssa.

Jos prosessissa lisäksi tapahtuu lämpötilamuutoksia voimme myös saada arvot pp m_s .:ssä että ϵ_0 :n lämpötilariippuvuudesta.

Patenttivaatimukset:

1. Menetelmä nesteen vesipitoisuuden mittausta varten, jossa menetelmässä mitataan nesteen ominaisuutta sähköisesti yhdellä parametrilla,

5 tunnettu siitä, että

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- nesteen ominaisuuksia mitataan ainakin likimain samanaikaisesti myös toisella sähköisellä menetelmällä, jolloin nesteen ominaisuuksia mitataan suhteellisella ja absoluuttimenetelmällä siten, että nesteestä mitataan dielektrisyysvakio sekä veden suhteellinen pitoisuus.
 - 2. Patenttivaatimuksen 1 mukainen menetelmä, tunnettu siitä, että mittaus toteutetaan kapasitiivisella anturilla.
 - 3. Jonkin edellisen patenttivaatimuksen mukainen menetelmä, t u n n e t t u siitä, että mittaus toistetaan kahdessa eri lämpötilassa niin nopeasti, että vesipitoisuuden voidaan olettaa pysyvän ainakin olennaisesti vakiosuuruisena.
- 4. Jonkin edellisen patenttivaatimuksen mukainen menetelmä, t u n n e t t u siitä, että nesteen vanhenemisesta aiheutuvia muutoksia vesipitoisuuden mittaukseen kompensoidaan käyttämällä vain tuoreinta mittaushistoriatietoa mittarin mittausarvon kompensointiin.
- 5. Jonkin edellisen patenttivaatimuksen mukainen menetelmä, tunnettu siitä, että nesteen, edullisesti öljyn ikääntymistä indikoidaan ε_0 :n muutoksista.
 - 6. Jonkin edellisen patenttivaatimuksen mukainen menetelmä, tunnettu siitä, että veden suhteellista pitoisuutta mitattaessa imeytetään mitattavassa nesteessä olevaa vettä apuväliaineeseen.
 - 7. Patenttivaatimuksen 6 mukainen menetelmä, t u n n e t t u siitä, että apuväliaine on polymeeriohutkalvo.

- 8. Patenttivaatimuksen 6 tai 7 mukainen menetelmä, tunnettu siitä, että apuväliaineen vesipitoisuus mitataan dielektrisyysvakion avulla.
- 9. Laitteisto nesteen vesipitoisuuden mittaamiseksi, joka laitteisto käsittää yhden sähköisen mittauselimen (5, 6 tai 1, 6) nesteen vesipitoisuuden mittaamiseksi,

tunnettu siitä, että

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- laitteistossa on myös toinen sähköinen mittauselin (1, 6 tai 5, 6) nesteen vesipitoisuuden mittaamiseksi, joka toinen mittauselin (1, 6 tai 5, 6) mittaa eri parametria kuin ensimmäinen sähköinen mittauselin (5, 6 tai 1, 6), jolloin mittauselimet ovat sellaiset, että toinen mittaa nesteen ominaisuuksia suhteellisella ja toinen absoluuttimenetelmällä siten, että toinen mittauselimistä (5, 6) on herkkä dielektrisyysvakion muutoksille ja toinen mittauselin on herkkä veden suhteelliselle pitoisuudelle.
- 10. Patenttivaatimuksen 9 mukainen laitteisto t u n n e t t u siitä, että dielektrisyysvakion muutoksille herkkä mittauselin (5, 6) on muodostettu kahdesta toistensa lomiin sijoitetusta sormielektrodista (5, 6).
 - 11. Patenttivaatimuksen 9 tai 10 mukainen laitteisto, tunnettu siitä, että toinen dielektrisyysvakion mittaukseen osallistuvista elektrodeista (6) muodostaa osan suhteellisen pitoisuuden mittaelektrodiparista (1, 6).
 - 12. Patenttivaatimuksen 9 mukainen laitteisto, t u n n e t t u siitä, että dielektrisyysvakion muutoksille herkkä mittauselin on muodostettu koaksiaalisesta rakenteesta, jossa toinen elektrodi muodostuu keskitapista ja vaippa on verkkomainen ja nestettä läpäisevä.
 - 13. Patenttivaatimuksen 9 mukainen laitteisto, t u n n e t t u siitä, että mittauselimet, jotka on sovitettu mittaamaan veden suhteellista pitoisuutta, sisältävät apuväliainetta, joka kykenee imeyttämään mitattavassa nesteessä olevaa vettä.

- 14. Patenttivaatimuksen 13 mukainen laitteisto, tunnettu siitä, että apuväliaine on polymeeriohutkalvo.
- 15. Patenttivaatimuksen 13 tai 14 mukainen laitteisto, t u n n e t t u siitä, että se käsittää elimet apuväliaineen dielektrisyysvakion määrittämiseksi apuväliaineen vesipitoisuuden määrittämistä varten.

(57) Tiivistelmä:

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Keksintö koskee menetelmää ja laitteistoa nesteen vesipitoisuuden mittausta varten. Menetelmän mitataan nesteen ominaisuutta sähköisesti yhdellä parametrilla. Keksinnön mukaan nesteen ominaisuuksia mitataan ainakin likimain samanaikaisesti myös toisella sähköisellä menetelmällä, jolloin nesteen ominaisuuksia mitataan suhteellisella ja absoluuttimenetelmällä.

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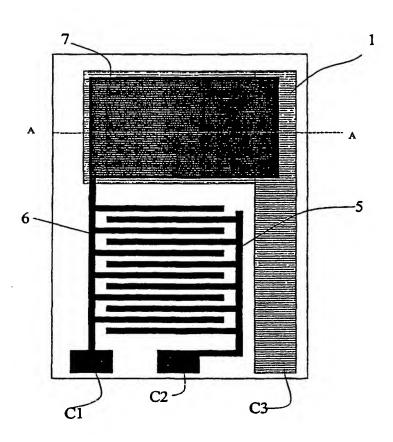
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(54) Title: METHOD AND APPARATUS FOR MEASURING WATER CONTENT



(57) Abstract: The invention relates to a method and apparatus for measuring the water content of a liquid. In the method, a property of the liquid is measured electrically for one parameter. According to the invention, the properties of the liquid are measured at least substantially simultaneously by another electrical method, too, whereby the properties of the liquid are measured using both a relative-value and an absolute-value measurement method.

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

● 1/PRTS

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METHOD AND APPARATUS FOR MEASURING WATER CONTENT

The invention relates to a method according to the preamble of claim 1 for measuring the water content of a liquid.

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The invention also relates to an apparatus for measuring the water content of a liquid.

US Pat. No. 5,331,287 describes a sensor, wherein interdigitated electrodes (finger electrodes) are coated with a conducting polymer. Water contained in the oil hydrates the polymer and thus modifies its conductivity. Also detects possible acids by way of protonation.

US Pat. No. 5,642,098 discloses a ring oscillator circuit, wherein electrical properties of the oil are measured with a number of measurement heads which measure the same parameter.

US Pat. No. 5,644,239 measures the electrical conductivity of a liquid (oil) at two elevated temperatures. The technique may be complemented with a possible optical measurement of oil opacity. A "figure of quality" may then be computed for the oil from these parameters.

US Pat. No. 5,656,767 describes a sensor system for measuring the change of an electrical parameter value (e.g., capacitance) in oil as a function of time. The same oil at a clean (dry) state may be used as a reference value. The same technique may be varied in multiple ways, e.g., by heating the oil sample.

Conventional techniques are handicapped in many aspects. Common methods for sensing absolute water volume content over the entire range of 0-100 % are the measurement of the dielectric coefficient and measurement of IR absorption. Both of these methods have in common that they require a zeroing step of the measurement system, whereby the reading must be reset to zero water content when the sensor is

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brought to measure an entirely dry (water-free) liquid. This step can be accomplished as a discrete zeroing operation or by using a sample of entirely dry oil in the sensor as a reference.

An additional complication arises therefrom that such a zero setting is typically dependent on the temperature.

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Also other factors besides the water content may affect the zero-value with the aging of the liquid.

Methods measuring the absolute water content are favored at high water contents (in the order of several per cent).

At lower water contents, problems generally arise from the marginal detection threshold and offset uncertainty (error of zero setting).

A relative value (aw) measurement method gives information on the water content value in relation to that of a fully saturated situation. However, a conversion to the volume percentage value of absolute water content remains undefined unless a conversion factor for the liquid being measured is known. The aw measurement method is suitable for use at low water content levels (nonsaturated and not emulsified), whereby the measurement has a sufficiently high sensitivity. Moreover, the method is free from zeroing problems.

It is an object of the present invention to overcome the drawbacks of the abovedescribed techniques and to provide an entirely novel type of method and apparatus for measuring the water content of a liquid.

The goal of the invention is achieved by way of measuring the water content of the oil/liquid using two different methods simultaneously, whereby the measurement technique is based on an absolute value measurement method complemented with a relative value measurement method.

More specifically, the method according to the invention is characterized by what is stated in the characterizing part of claim 1.

Furthermore, the apparatus according to the method is characterized by what is stated in the characterizing part of claim 9.

The invention offers significant benefits.

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The combination of an absolute value measurement method with a relative value measurement method (aw-type of measurement) makes it possible to eliminate the need for a zeroing step in the absolute value measurement.

By way of performing a sequence of rapidly repeated measurements at different temperatures, it is also possible to eliminate errors caused by temperature variations.

In the following, the invention will be examined in more detail by means of exemplifying embodiments illustrated in the appended drawings, in which:

20 <u>FIG. 1 shows a top view of an embodiment of an electrode structure according to the invention; and</u>

FIG. 2 shows a sectional view of the sensor structure of FIG. 1 along line A-A.

As shown in FIGS. 1 and 2, the embodiment according to the invention can be implemented using a structure, wherein onto the surface of a substrate 4 is formed a combination of three electrodes. The surface of the substrate 4 has directly deposited thereon a pair 3 of bottom electrodes formed by electrodes 5 and 6. In the illustrated case, the electrodes are shaped into finger electrodes, more specifically disposed as interdigitated electrodes, whereby the interelectrode surface is maximized by using an electrode structure having the finger electrodes displaced between each other. The length of the adjacent edges of the interdigitated electrodes 5 and 6 is equivalent to

the area of superimposed electrodes in a planar capacitor. The width and interelectrode gap of the electrodes 5 and 6 typically are in the range of 5 to 500 micrometers. In addition to its interdigitated finger electrode portion, the electrode 6 has a planar area 7. Furthermore, the electrodes 5 and 6 include contact pad areas C1 and C2 for connection of the electrodes to measurement means. Onto the bottom electrode pair 3 is deposited a polymer layer 2 having a thickness of 0.5-5 micrometer typical. The area of the polymer layer 2 may also be extended over the interdigitated finger electrode structure 5 and 6, whereby it also functions as a passivation layer and reduces the effect of conducting particles contained in the liquid sample on the measurement result. Onto the polymer layer 2 is deposited a water-permeable top electrode 1 with a contact pad area C3 for external connections. The top electrode 1 is aligned above the rectangular solid area 7 of the electrode 6 in order to form a planar capacitor structure.

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The structure illustrated in FIGS. 1 and 2 is utilized as follows. The dielectric coefficient of the liquid under measurement is measured over the contacts C1 and C2 of the electrodes 5 and 6. Respectively, the relative value measurement is performed over the electrodes 6 and 1, that is from the contacts C1 and C3.

Advantageously, the liquid whose water content is measured is oil, but the water content measurement according to the invention may also be performed on other liquids such as a hydraulic fluid, gasoline or a coolant as well.

According to the invention, the measurement of the activity of water can be carried out as follows.

Firstly, it must be noted that the water activity measurement is only an exemplifying embodiment of the relative value measurement technique according to the invention.

As shown in FIGS. 1 and 2, the sensor may comprise a polymer layer 2 deposited between two electrodes as an element, whose absorbency of water is a function of the activity of water in its immediate environment. Typically, such sensors are used

as relative humidity transducers, for example. This type of measurement method is characterized in that the measurement result indicates the degree of water activity, that is:

$$5 aw = F(ppm/ppm_s(T)), (1)$$

where

ppm = volumetric proportion of water in solution×10⁶

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ppm_s = volumetric proportion of water in saturated solution×10⁶

The function F may for example be assumed to have a linear nature, whereby:

$$aw = ppm/ppm_s(T)$$
 (2)

The value of aw varies from 0 (entirely dry liquid) to 1 (entirely saturated water solution).

Hence, a mere water activity measurement gives information on how close the situation is either to an emulsified state or a water-separation state. However, when the state of emulsification or water separation is reached, the value of water activity becomes very close to 1, whereby no information on the state of the liquid can be obtained. Nevertheless, the method can render at values of aw < 0.9 in a very sensitive manner such oil/liquid-independent information that, for example, is related to the lubrication properties of the measured sample.

At room temperature, the value of ppm_s may vary from 20 ppm for a basic oil having no additives to values above 10,000 ppm for lubrication oils rich with additives.

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Next, the measurement technique of the dielectric coefficient is elucidated in more detail. The measurement of the dielectric coefficient is only one exemplifying

embodiment of the absolute-value concentration measurements possible within the scope of the invention.

In the measurement of the dielectric coefficient of a liquid, the sensor may be implemented either using the interdigitated electrode (finger-electrode) structure according to FIGS. 1 and 2 or, alternatively, formed into a coaxial pair of electrodes brought into contact with the liquid to be measured. The output signal of the sensor is dependent on both the dielectric coefficient and water content of the liquid under measurement:

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$$\varepsilon_{\rm r} = \varepsilon_0 + {\rm F(ppm)} \tag{3}$$

where

15 ε_0 = dielectric coefficient of entirely dry liquid,

F(ppm) = a function dependent on the water content. Over a limited range of water content, the function may be assumed to be linear, that is:

$$20 \varepsilon_{\rm r} = \varepsilon_0 + a \times \rm ppm (4)$$

where

a = constant independent of the liquid type.

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An advantage of the dielectric coefficient measurement technique is that it covers the entire possible measurement range from 0 to 100 vol-% water. A disadvantage of the method is that its sensitivity at the low end of water content (where the greatest interest usually lies) is low and that the value of ε_0 must be known. Typically, the measurement system is calibrated using dehydrated oil as a standard.

If both of the above-described measurement techniques are employed simultaneously, a number of different calibration techniques can be used for a given liquid.

If the liquid is known by the value of its ppm_s(T), the following approach is possible:

First, the values of aw and ε_r are measured simultaneously. If aw is less than 1, formulas 2 and 4 may be assumed to be valid.

Then, the combination of formulas 2 and 4 can be solved for ε_0 and ppm. It is also possible to estimate the value of ppm_s(T) if the type of the liquid is known, whereby the result thus obtained, however, remains slightly more inaccurate.

If the value of ppm_s(T) of the liquid is unknown, the following procedure can be carried out:

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The sensor output is measured at two (unknown) water content values. If the value of aw is smaller than 1 in both cases, an equation group of four equations and four unknown variables is obtained, which means the equations can be solved in unique manner. Assigning subindices 1 and 2 for the measurement results of the two sessions, respectively, the following formula can be written:

$$\varepsilon_0 = (aw_2 \times \varepsilon_{r1} - aw_1 \times \varepsilon_{r2})/(aw_2 - aw_1)$$
 (5)

This procedure may also be arranged to take place automatically during the continuous function of the measurement apparatus if the water content of the liquid under measurement varies.

If the measurement data is collected by more than two pairs of values, the unknown terms can be fitted to the data using, e.g., the least squares method.

The benefit of the latter technique is that possible changes in the values of ε_0 or ppm_s(T) due to temperature variations or aging/soiling of the liquid can be compensated for. In fact, the changes detected in ε_0 serve as an indicator of aging in oil.

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If the temperature of an oil sample or, respectively, of a sample flow, is changed so rapidly that the water content of the sample can be assumed to stay substantially unchanged, it is possible to determine the temperature dependence of the dielectric coefficient of an entirely dry liquid by way of measuring essentially simultaneously the ε_r and temperature of the liquid at at least two temperatures.

For example it can be assumed assuming that ε_0 is a linear function with temperature:

$$\varepsilon_{\rm r} = b0 + b1 \times T + a \times ppm \tag{6}$$

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whereby the following formula can be formed

$$\varepsilon_{r}(T2) - \varepsilon_{r}(T2) = b1 \times (T2 - T1) \tag{7}$$

wherefrom the coefficient b1 can be solved. Also in this case, it is possible to collect values at a greater number of temperatures and then fit the measurement results with the help of the least squares method. This technique gives a continuously reliable parameter estimate value for ε_0 as the temperature varies.

Also the temperature dependence of ppm_s(T) can be determined simultaneously.

Over a limited temperature range, a general assumption may be made as:

$$ppm_s(T) = c0 \times e^{(C1 \times T)}$$
(8)

30 where

 $c0 = value of ppm_s at T = 0 °C$

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c1 = coefficient of temperature dependence.

Then, the value of c1 can be solved by way of measuring aw at at least two temperatures with the assumption that the water content of the liquid remains unchanged during the measurements:

$$c1 = LN(aw1/aw2)/(T2-T1)$$
 (9)

Advantageously, these two determinations may be carried out simultaneously.

According to a preferred embodiment of the invention, the zeroing operation is performed automatically each time the measurement of aw gives a sufficiently low value. The lower the value of aw the more accurate the zeroing operation. The uncertainty of the method is associated with the value of ppm_s(T) that is dependent on the type of liquid under measurement and thus can be obtained by an "intelligent guess".

According to another preferred embodiment of the invention, two samples of the liquid/oil having different water contents are taken. Then, the measurement system may intentionally be set to measure two samples of different water contents or, alternatively, gradual accumulation of data from the measured process is utilized, whereby the natural variation of water content in the monitored process is availed of. This approach also gives a value for ppm_s(T) on the basis of which it is possible according to the invention to compute from the measured value of aw the correct value of ppm without resorting to an "intelligent guess".

If a continuous data collection from the monitored process is performed using simultaneously a sliding-window technique for "dumping" obsolete data, it is also possible to compensate for changes in both the value of ppm_s and ϵ_0 due to aging of the liquid/oil.

In the case that the process also is subjected to temperature changes, the temperature dependencies of ppm_s and ϵ_0 can also be resolved.

What is claimed is:

1. Method for measurement of water content of a liquid, in which method a property of the liquid is measured electrically for one parameter,

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- the properties of the liquid are measured at least substantially simultaneously also by another electrical method, whereby the properties of the liquid are measured using both a relative-value measurement method and an absolute-value measurement method resulting in the measurement of the liquid for its dielectric coefficient and relative water content

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characterized in that

 said measurement is repeated at two different temperatures in a so rapid succession that the water content of the liquid may be assumed to stay at least substantially constant.

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- 2. Method according to claim 1, c h a r a c t e r i z e d in that the temperature dependence of the dielectric coefficient of an entirely dry liquid is determined by measuring essentially simultaneously the ε_r and temperature of the liquid at at least two temperatures.
- 3. Method according to claim 1 or 2, c h a r a c t e r i z e d in that said relative water content measurement is carried out using a capacitive sensor.

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4. Method according to any one of foregoing claims, c h a r a c t e r i z e d in that changes in the results of water content measurement due to aging of the liquid are compensated for by virtue of using only the most recent data of the measurement history for the compensation for changes in the response of the measurement system.

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- 5. Method according to any one of foregoing claims, c h a r a c t e r i z e d in that the aging of said liquid, advantageously oil, is indicated on the basis of changes in the value of ε_0 .
- 6. Method according to any one of foregoing claims, characterized in that, in the measurement of relative water content, an auxiliary medium is used for absorbing thereto the water contained in the liquid under measurement.
- 7. Method according to claim 5, characterized in that said auxiliary medium is a thin-film polymer layer.
 - 8. Method according to claim 5 or 6, c h a r a c t e r i z e d in that the water content of said auxiliary medium is determined by way of measuring its dielectric coefficient.
- 9. Apparatus for measurement of the water content of a liquid, said apparatus comprising one electrical sensor means (5, 6 or 1, 6) for measuring the water content of a liquid, which apparatus includes
 - a second electrical sensor means (1, 6 or 5, 6) for measuring the water content of a liquid, said second sensor means (1, 6 or 5, 6) measuring a different parameter than that measured by said first electrical sensor means (5, 6 or 1, 6), said sensor means being such that one of them measures the properties of the liquid by a relative-value measurement method and the other by an absolute-value measurement method, whereby one sensor means (5, 6) is sensitive to changes in the dielectric coefficient and the other sensor means is sensitive to the relative water content,

characterized in that

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- the sensor means adapted for measuring the relative water content contain an auxiliary medium capable of absorbing water contained in the liquid under measurement.
- 10. Apparatus according to claim 9, c h a r a c t e r i z e d in that said sensor means (5, 6) sensitive to changes in dielectric coefficient is formed by two interdigitated finger electrodes (5, 6).
- 11. Apparatus according to claim 9 or 10, c h a r a c t e r i z e d in that one electrode

 (6) of the sensor pair adapted to perform the measurement of dielectric coefficient

 also forms a part of the measurement electrode pair (1, 6) adapted to perform the

 measurement of the relative water content.
 - 12. Apparatus according to claim 9, c h a r a c t e r i z e d in that the sensor means sensitive to changes in the dielectric coefficient is formed by a coaxial structure, wherein one electrode is formed by a center pin and the jacket has a net-like structure and is permeable to water.
 - 13. Apparatus according to claim 12, characterized in that said auxiliary medium is a thin-film polymer layer.

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14. Apparatus according to claim 12 or 13, c h a r a c t e r i z e d in that the apparatus contains means adapted to measure the dielectric coefficient of said auxiliary medium whereupon the relative water content of said auxiliary medium can be determined.

[57] Abstract

The invention relates to a method and apparatus for the measuring the water content of a liquid. In the method, a property of the liquid is measured electrically for one parameter. According to the invention, the properties of the liquid are measured at least substantially simultaneously by another electrical method, too, whereby the properties of the liquid are measured using both a relative-value and an absolute-value measurement method.

(FIG. 1)

International application No. PCT/FI 00/00542

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: G01N 27/02, G01N 27/22, G01N 33/28
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: GO1N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPODOC, WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Y Further documents are listed in the continuation of Box C.

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| * | Special categories of cited documents: | "T" | later document published after the international filing date or priority |
|-----|---|-------|--|
| "A" | document defining the general state of the art which is not considered to be of particular relevance | | date and not in conflict with the application but cited to understand the principle or theory underlying the invention |
| "E" | ertier document but published on or after the international filing date | "X" | document of particular relevance: the claimed invention cannot be |
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| | special reason (as specified) | "Y" | document of particular relevance: the claimed invention cannot be |
| .0. | document referring to an oral disclosure, use, exhibition or other means | | considered to involve an inventive step when the document is combined with one or more other such documents, such combination |
| "P" | document published prior to the international filing date but later than | | being obvious to a person skilled in the art |
| | the micrity date claimed | # D.# | de marie de la combinación de la companya del companya de la companya de la companya del companya de la company |

X See patent family annex.

| | - comment intended of the same patent family |
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| Date of the actual completion of the international search | Date of mailing of the international search report |
| | 26 October 2000 (26.10.00) |
| 23 October 2000 | |
| Name and mailing address of the ISA/ | Authorized officer |
| Swedish Patent Office Box 5055, S-102 42 STOCKHOLM | Jonas Andersson/GH |
| Facsimile No. +46 8 666 02 86 | Telephone No. +46 8 782 25 00 |

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03/10/00

International application No.
PCT/FI 00/00542

| | ent document in search report | | Publication date | Patent family member(s) | | Publication date |
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